Technical Review

Advanced Battery Materials Analysis

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Abstract

Secondary batteries are essential devices for achieving a carbon-neutral society, and demand for them is growing significantly for electric vehicles. Development of next-generation batteries aimed at increasing battery capacity and improving recyclability is accelerating. Nippon Steel Technology Co., Ltd. has established a one-stop solution system combining battery materials composition and structural analysis with battery prototyping, battery performance evaluation, and safety testing.

1. Introduction

1.1 Background

Various initiatives are underway globally to achieve a carbon-neutral society by 2050. Battery energy storage system are being explored to reduce fossil fuel consumption and effectively utilize renewable energy. Lithium-ion batteries (LIBs) using organic liquid electrolytes are currently the mainstream secondary batteries used in these system. However, from a safety perspective, all-solid-state lithium-ion batteries (all-solid-state LIBs) using sulfide solid electrolyte are also being actively researched and developed. Furthermore, batteries such as sodium-ion batteries and lithium-sulfur batteries are being researched as future options. To enhance the performance of these secondary batteries, the chemical composition and crystal structure of the battery materials used must be strictly controlled. Consequently, analysis and evaluation of not only battery materials manufactured from new raw materials but also recycled battery materials produced from recycled raw materials have become crucial,[1] necessitating a comprehensive, end-to-end evaluation from materials to battery performance.

1.2 Nippon Steel Technology Co., Ltd.'s approach to battery material evaluation and analysis

Figure 1 shows an evaluation flow combining battery material assessment with battery performance and safety testing. We are aiming to establish a framework for a one-stop solution that handles everything from materials to product evaluation. This enables us to re-

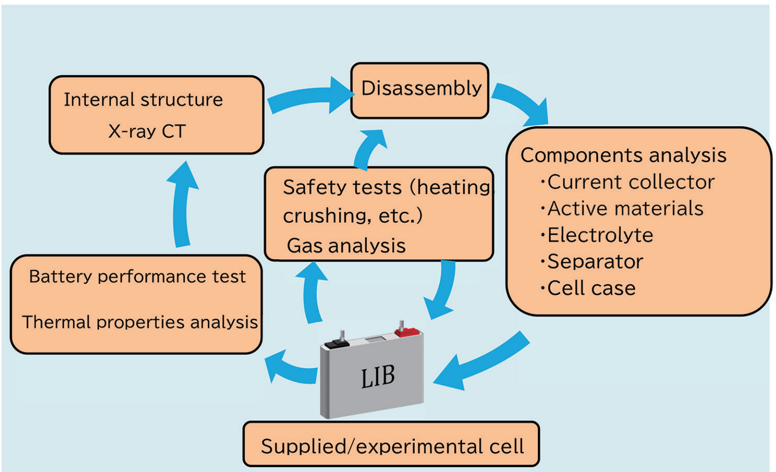

Fig. 1 Test flowchart from material evaluation to battery performance analysis

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spond to the needs of a wide range of customers, including material manufacturers, battery manufacturers, and battery users, and to build a framework capable of handling everything from degradation investigations of existing batteries to supporting the research and development of next-generation batteries.

2. Case Study of One-stop Solution

2.1 Analysis and evaluation technology menu

Table 1 lists evaluation items for battery materials and components. We apply conventional steel material analysis and evaluation techniques to assess various battery materials. Secondary batteries utilize lithium and sodium, which are reactive to atmospheric moisture and oxygen, necessitating handling in non-exposed to the air. Samples are retrieved inside an Ar glove box (dew point <-90°C) or dry booth (dew point <-60°C), then transferred via transfer vessels to various analytical instruments to avoid material degradation during analysis and evaluation.

Table 2 lists various physical property evaluation items and their measurement devices. During battery operation, electric current generates heat, making electrical and thermal characteristics crucial physical properties. These serve as fundamental information for simulation-based battery pack design. These physical property val-

Table 1 Evaluation equipment/methods for battery materials

Components	Objects	Evaluation items	Evaluation equipment/methods
Electrode (Anode/Cathode)	Active material	Chemical composition, crystal structure, morphology	XPS, TEM, AES, SEM, XAFS Raman, TG-DTA, DSC, SPM, ESR
	Conductive additive	Crystal structure	XRD, TEM, Raman, SPM
	Binder	Chemical species, content	FT-IR, GC-MS, XPS, SPM
	SEI	Chemical species, molecular structure, morphology	XPS, AES, HAXPES, SPM, TOF-SIMS, TEM, SEM, GD-OES
Separator	Material	Pore morphology/distribution, thermal properties	SEM, FT-IR, GC-MS, SPM, mercury porosimeter, TG-DTA, DSC, TMA
Current collector	Foils	Corrosion resistance, electric conductivity	Electrochemical test, XPS, AES, GD-OES
Tab lead	Sheet/foil	Strength, corrosion resistance, weldability	Tensile test, corrosion test, welding test
Electrolyte	Solvent Electrolyte	Chemical species, composition	GC-MS, NMR, FT-IR, ICP
Case	Cell	Mechanical strength, corrosion resistance	Static/dynamic strength test, crush test, corrosion test
	Pack		
Battery/Module	Internal structure	Physical structure	X-ray CT, disassembly

Table 2 Physical property evaluation techniques for battery materials

Properties	Analysis items	Measurement method	Measurement range
Thermal properties	Thermal conductivity	Laser flush analysis	$-100\sim1400$°C
		Thermal gradient method	$-30\sim700$°C
		Hot wire method	RT~1300°C
	Specific heat capacity	Drop calorimeter for high-temperature	$600\sim1500$°C
		Adiabatic specific heat measurement	$100\sim800$°C
		DSC (differential scanning calorimetry)	$100\sim1400$°C
		DSC (differential scanning calorimetry)	$-100\sim500$°C
	Thermal analysis	TG-DTA (-MS) (Thermogravimetric-Differential thermal analysis (-mass spectroscopy))	RT~1600°C
	Thermal expansion	Push-rod dilatometer	RT~1700°C
		High-precision thermal dilatometer	$-180\sim500$°C
Powder properties Mechanical properties	Modulus	Resonance method	$-170\sim1200$°C
	Young's modulus	JIS method	RT~1100°C
	Powder properties Porus component	Powder size distribution analysis	$0.02\sim1000$ μm
		Specific surface area measurement	$0.001\sim500$ m^2/g
		Pore size distribution	6 nm$\sim$400 μm
		Bulk density/true density	min 0.1 cc
Electrical properties	Electrical resistance	Four-terminal sensing	RT~1000°C vac
		High resistance with low current test	RT~1000°C air
	Breakdown voltage	Dielectric breakdown test	Max 10 kV
	AC impedance	LCR meter using frequency sweep	20 Hz$\sim$2 MHz

ues can be obtained for individual battery materials or in combined states.

2.2 Chemical analysis of battery materials

Cathode materials contain Ni, Mn, and Co, and their atomic ratios significantly affect battery capacity. Accurate analysis of composition ratios is essential during material recycling. **Table 3** shows analysis results for cathode powder samples and cathode compound samples collected from a prototype cathode made using that powder. Composition ratios were accurately measured, enabling quality verification of cathode materials using recycled raw materials.[2] The crystal structure of cathode materials can be analyzed using X-ray diffraction (XRD). Furthermore, impurities in cathode materials, such as Li_2CO_3 and LiOH, can be quantified using potentiometric titration.

Carbon materials are used for anode materials and as conductive additives to enhance inter-particle conductivity. Nippon Steel Technology possesses expertise cultivated through analyzing high-purity graphite used in nuclear power and semiconductor fields. **Table 4** shows the results of analyzing various battery-grade carbon materials. This demonstrates the capability to precisely measure the types and quantities of impurity elements in carbon materials, enabling their utilization in developing batteries with excellent long-term durability.

2.3 Battery prototyping and battery performance evaluation

By combining various battery materials to prototype batteries and evaluating their performance, the correlation between material properties and battery performance can be evaluated. Since LIBs contain Li, which is reactive to air, battery fabrication is performed in a glove box (**Fig. 2**) or dry booth (**Fig. 3**). Particularly for all-solid-state LIBs, sulfied-based solid electrolytes readily react with moisture, necessitating a lower dew point environment than liquid LIBs. Therefore, we have established a dedicated glove box for all-solid-state LIBs to fabricate prototype batteries. These glove boxes are also utilized for material analysis and characterization. Batteries fabricated here undergo charge-discharge testing in a constant-temperature chamber to evaluate battery performance as shown in **Table 5**.

2.4 Evaluation of physical properties of electrodes

Battery electrodes are fabricated by coating slurry made from powdered electrode materials and organic binders onto metal foil. Since the adhesion strength affects battery life, the SAICAS (Surface And Interfacial Cutting Analysis System) shown in **Fig. 4** (a) is used to physically measure this adhesion strength. While this technology is also used for resin coating inspections on painted steel sheets, it was newly introduced into the dry booth specifically for battery evaluation, enabling measurements under non-exposed atmospheric conditions (dew point <-60°C). Figure 4 (b) shows a comparison of the adhesion properties between electrodes. Differences in adhesion can be quantitatively evaluated as differences in horizontal cutting force.[3]

Other electrode properties measurable include specific surface area and pore size distribution. Comparing these electrode property values with battery charge/discharge battery test results allows investigation of the influence of electrode structure on battery performance.

2.5 Thermal property evaluation of batteries

Temperature control is necessary during battery use to prevent overheating or temperature drop, requiring thermal property measurements. **Table 6** shows thermal conductivity measurement methods. Thermal properties of battery materials and battery cells can be measured using the temperature gradient method shown in **Fig. 5**.[4] Thermal property values obtained by such methods are used in heat

Table 3 Cathode material analysis

Specimen	Unit	Ni	Mn	Co	C
Active material	mass%	30.3	17.1	12.2	0.03
	atomic ratio	5.00	3.00	2.00	***
Electrode mixture*	mass%	28.3	15.8	11.4	4.63
	atomic ratio	5.00	3.00	2.00	***
*Electrode mixture ratio		NMC532 : AB : PVdF = 93 : 3 : 4 (mass%)			

Table 4 Anode materials analysis

ppm (mg/kg)

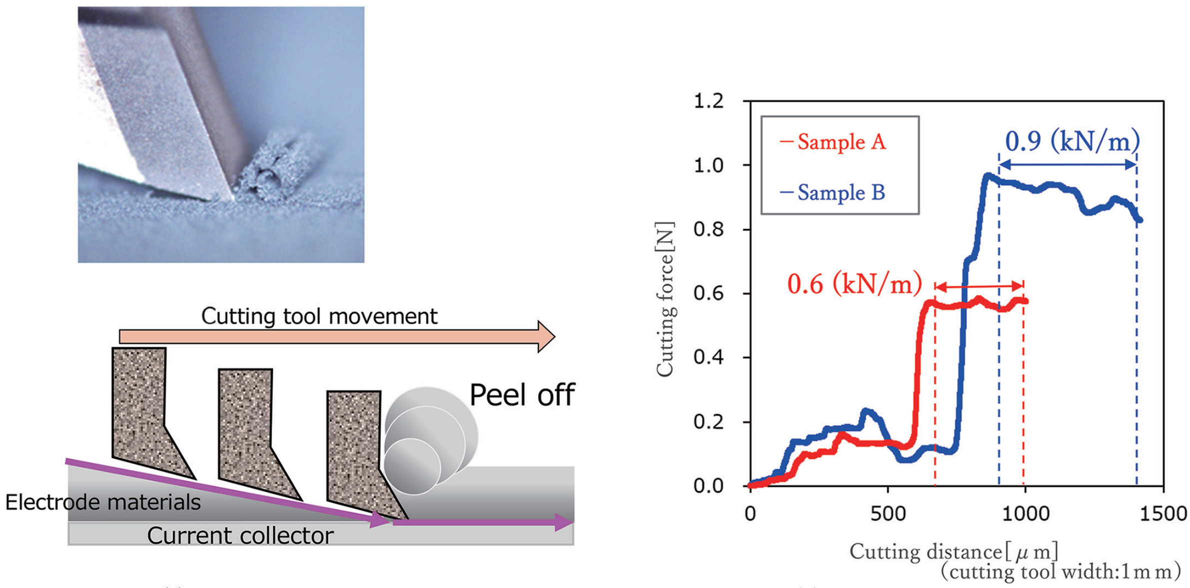
Specimen		Li	Na	Al	Ni	Mn	Co	Fe	Cr
Conductive additive (Acetylene black)	Standard purity	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	1.9	<0.1
	High purity	<0.1	<0.1	0.2	<0.1	<0.1	<0.1	0.4	<0.1
Anode material (Graphite)	Natural	<0.1	4.9	3.5	0.5	0.5	<0.1	14.5	1.9
	Artificial	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

Fig. 2 Globe box for LIB prototyping (DP <-90°C)

Fig. 3 Dry booth for LIB materials analysis (DP <-60°C)

Table 5 Evaluation items and test methods for battery characteristics

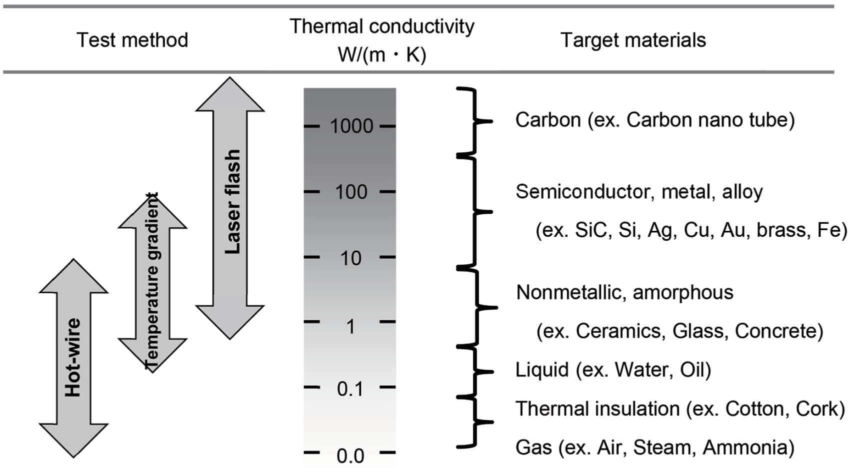
Battery characteristics	Test method	Evaluation items
Electrical capacity	Charge and discharge test	Cell capacity Electrical efficiency High-current performance Temperature dependence
Cycle lifetime	Cycle test and Degraded cell disassembly	Capacity decay Electrode expansion Cell expansion
Corrosion resistance	Linear-scan voltammetry potentiostatic test	Reaction voltage Dissolution amount
Erectrical impedance/resistance (Surface reaction products formation)	AC impedance DCR measurement X-ray CT	Internal resistance (AC/DC) Analysis of surface products Internal structure



(a)SAICAS measurement schematic (b) Adhesion strength testing

Fig. 4 SAICAS for battery electrode adhesion test

Table 6 Thermal conductivity test method



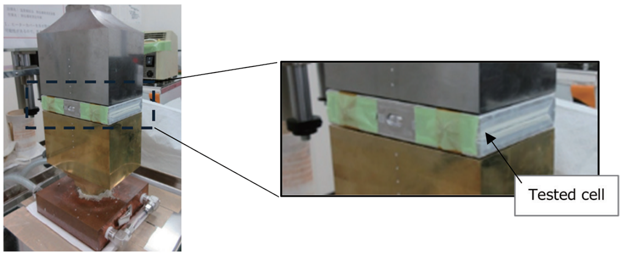


Fig. 5 Measurement of battery thermal conductivity using the temperature gradient method

transfer simulations of battery cells and battery packs, aiding in the development of battery packs with excellent heat transfer and heating performance.

2.6 Quantitative analysis of metal Li deposited on the anode

Metal Li deposited on the LIB negative electrode during charge-discharge battery testing causes capacity degradation and reduced safety due to internal short circuits. After degradation, the negative electrode can be removed in non-atmospheric environment glove box, and the amount of deposited metal Li can be quantified using an electron spin resonance (ESR) device.[5] Li deposition does not occur uniformly across the electrode surface but occurs locally where electrolyte depletion or changes in electrode spacing occur. ESR, which can measure small-area samples, is characterized by its ability to quantify such Li deposition. **Figure 6** and **Table 7** show the ESR measurement results for samples collected from a liquid LIB degraded by rapid charge-discharge in a non-atmospheric environment. The white areas confirm significant Li metal deposition, and its quantity can be quantified.

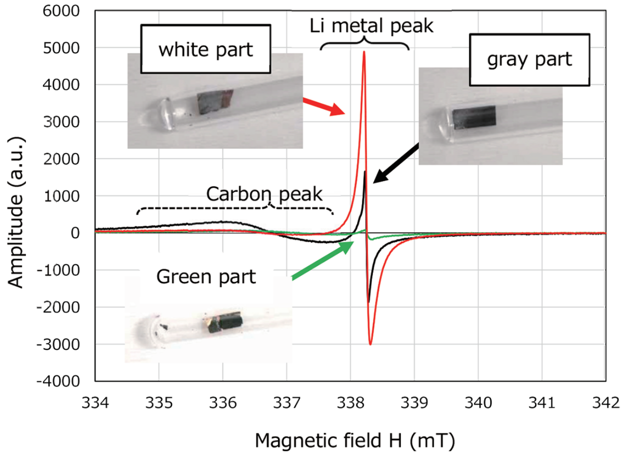


Fig. 6 Analysis of metallic-Li deposition on degraded LIB anode

Table 7 Quantitative calculation result

	Li metal	
Sample	(ng)	(ng/mm^2)
White	30.08*	1.97*
Gray	5.35	0.28
Green	1.09	0.08

* Quantitative values were underestimated due to the skin effect.

2.7 Safety testing

(1) Battery thermal propagation test

If one cell ignites within a battery pack containing multiple cells and the fire spreads to adjacent cells, the entire battery pack can become engulfed in flames, creating a dangerous situation. **Figure 7** shows the appearance of cells after triggering a thermal runaway in one cell and spreading the fire to adjacent cells. It was confirmed that using high-melting-point steel instead of low-melting-point aluminum for the cell case can double the cell fire propagation time.[6] After the test, the Al cell case sidewall was melted, indicating high-temperature combustion gases may have reached adjacent cells directly. However, the steel cell case remained unmelted, with high-temperature combustion gases/solids released only through the safety vent. Using steel cell cases may allow control of the flow of combustion gases/solids released through the safety vent, potentially preventing thermal spread. This suggests steel cell cases can enhance battery pack safety.

(a) Ni-coated steel sheet (b) Aluminum sheet

Fig. 7 Appearances of lithium-ion batteries after thermal runaway propagation test

(2) Battery crush test and continuous released gas analysis

A crush test machine transportable into the explosion-proof test chamber was used to crush a fully charged battery pack. The resulting released organic gas components were analyzed in real-time using a vacuum ultraviolet-single photon ionization-time-of-flight mass spectrometer (VUV-SPI-TOFMS).[7] This technology also utilizes evaluation techniques from the steel industry.[8] **Figure 8** shows the measurement results of gases emitted from the crushed and combusted battery pack in a fully charged state. The crushing of the battery caused ignition and smoke generation, and the internal temperature of the battery pack rose to approximately 900°C. Simultaneously with the temperature rise, carbon monoxide, benzene, and toluene were generated, followed by naphthalene and, later, phenol gases. Thus, this device enables real-time gas analysis, providing test data useful for elucidating the battery combustion mechanism. Furthermore, this measuring device is a compact, lightweight, vehicle-mounted portable type that can be moved to the test site for measurement.

3. Conclusion

Nippon Steel Technology has established an evaluation system for evaluating battery materials from various perspectives by combining battery prototyping, battery performance evaluation, and safety testing technologies with various analytical and analysis technologies that have been used in the steel industry. The development of next-generation batteries aimed at increasing battery capacity and improving recyclability is accelerating, and we aim to provide a one-stop solution that meets customer needs with the evaluation

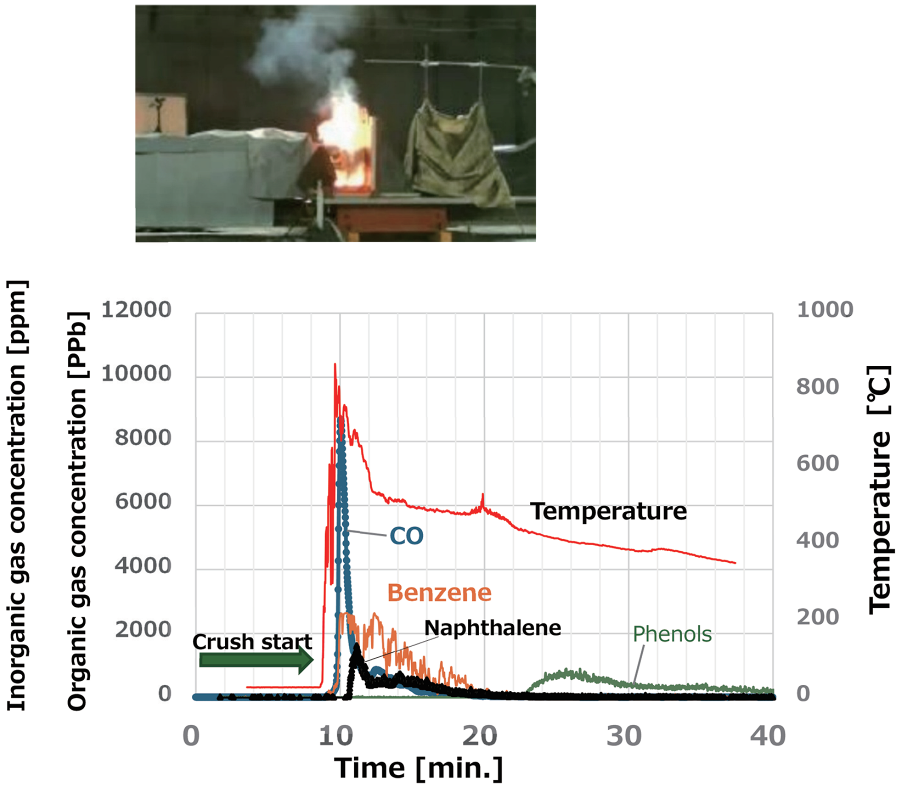


Fig. 8 Battery pack compression failure test and real-time gas analysis

technology described in this report. For details on the cases introduced, please refer to the following references.

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